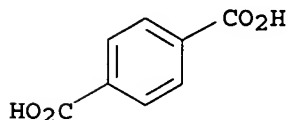


=> s 100-21-0/rn  
L1 1 100-21-0/RN

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L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2006 ACS on STN  
RN 100-21-0 REGISTRY  
ED Entered STN: 16 Nov 1984  
CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)  
OTHER CA INDEX NAMES:  
CN Terephthalic acid (7CI, 8CI)  
OTHER NAMES:  
CN 1,4-Dicarboxybenzene  
CN 4-Carboxybenzoic acid  
CN NSC 36973  
CN p-Benzenedicarboxylic acid  
CN p-Carboxybenzoic acid  
CN p-Dicarboxybenzene  
CN p-Phthalic acid  
CN TA 33LP  
CN TPA  
CN WR 16262  
FS 3D CONCORD  
DR 211863-90-0, 211863-92-2  
MF C8 H6 O4  
CI COM  
LC STN Files: ADISNEWS, AGRICOLA, ANABSTR, AQUIRE, BEILSTEIN\*, BIOSIS,  
BIOTECHNO, CA, CAOLD, CAPLUS, CASREACT, CBNB, CHEMCATS, CHEMINFORMRX,  
CHEMLIST, CIN, CSChem, CSNB, DDFU, DETHERM\*, DRUGU, EMBASE, ENCOMPLIT,  
ENCOMPLIT2, ENCOMPAT, ENCOMPAT2, GMELIN\*, HSDB\*, IFICDB, IFIPAT,  
IFIUDB, IPA, MEDLINE, MRCK\*, MSDS-OHS, NAPRALERT, PIRA, PROMT, RTECS\*,  
SPECINFO, SYNTHLINE, TOXCENTER, TULSA, ULIDAT, USPAT2, USPATFULL, VTB  
(\*File contains numerically searchable property data)  
Other Sources: DSL\*\*, EINECS\*\*, TSCA\*\*  
(\*\*Enter CHEMLIST File for up-to-date regulatory information)



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

9793 REFERENCES IN FILE CA (1907 TO DATE)  
1992 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA  
9820 REFERENCES IN FILE CAPLUS (1907 TO DATE)  
1 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

=> file caplus  
COST IN U.S. DOLLARS  
FULL ESTIMATED COST

SINCE FILE	TOTAL
ENTRY	SESSION
2.34	2.55

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FILE LAST UPDATED: 9 Jun 2006 (20060609/ED)

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L7      18 L6 AND PY<2004
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L7  ANSWER 1 OF 18  CAPLUS  COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:      2004:942990  CAPLUS
DOCUMENT NUMBER:      143:121166
TITLE:                Environmentally friendly liquid phase oxidation of
                        p-xylene using SBA-15 supported transition metal ions
AUTHOR(S):            Kim, Yong-Ho; Son, Young-Bae
CORPORATE SOURCE:      Research Institute of Industrial Technology, College
                        of Engineering, Chungnam National University, Daejeon,
                        S. Korea
SOURCE:                Nonmunjip - Ch'ungnam Taehakkyo Sanop Kisul Yon'guso (
                        2003), 18(2), 92-103
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PUBLISHER: Ch'unghnam Taehakkyo Sanop Kisul Yon'guso  
DOCUMENT TYPE: Journal  
LANGUAGE: Korean

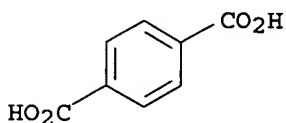
AB Com. processes for homogeneous liquid phase oxidation of p-xylene are in need of the use of complex reaction system like catalytic metal ions, corrosive bromide ion, and acidic solvent. Replacement of the homogeneous catalyst with a heterogeneous catalyst and exclusion of bromide ion are very desirable, because which will eliminate toxic metal and bromide ion from the waste effluent of the process. As a such alternative, active transition metal species (Co, Mn, Ni, Fe, or Cu) supported on a chemical modified SBA 15 silica were prepared and used as heterogeneous catalysts for the environmentally friendly liquid phase oxidation of p-xylene. The Co species bound on the SBA 15 (Co-SBA 15) was found to be the most effective for the oxidation of p-xylene to aromatic carboxylic

acids among other transition metal species. The effect of reaction variables such as reaction time (0.5-9 h), temperature (130-190°), total pressure (10-25 atm), and the partial pressure of oxygen (1-9 atm) was studied with the Co-SBA 15 catalyst. At high levels of conversion, the considerable catalytic activities to p-toluic acid were observed, even though formation of terephthalic acid, an solid product, was very low and then the catalyst was easily deactivated due to build-up of solid products on active sites. The supposed reaction network based on the selectivities of partial oxidation products was the analogy compared with that on the homogeneous Co/Mn/Br catalyst. This indicates that the Co-SBA 15 catalyst, a heterogeneous catalyst which can behave like homogeneous catalysts, may be well applied for the liquid phase oxidns. without the formation of solid products.

IT 100-21-0, Terephthalic acid, processes  
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PROC (Process)  
(environment friendly liquid phase oxidation of p-xylene using silica supported transition metal ions)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 2 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2002:845607 CAPLUS

DOCUMENT NUMBER: 137:338386

TITLE: Method and catalyst system for preparing aromatic carboxylic acids from alkylaromatics by liquid-phase oxidation

INVENTOR(S): Park, Sang-Eon; Yoo, Jin S.; Jun, Ki-Won; Raju, David B.; Kim, Young-Ho

PATENT ASSIGNEE(S): Korea Institute of Chemical Technology, S. Korea

SOURCE: U.S., 6 pp.  
CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

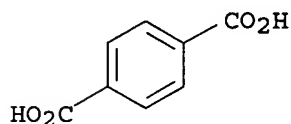
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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US 6476257	B1	20021105	US 2001-964750	20010928 <--
US 2002193631	A1	20021219		
KR 2002076879	A	20021011	KR 2001-17072	20010331 <--
JP 2002332255	A2	20021122	JP 2002-94095	20020329 <--
PRIORITY APPLN. INFO.:			KR 2001-17072	A 20010331

AB In the title process, aromatic carboxylic acids (e.g., terephthalic acid) are prepared from alkylarom. hydrocarbons (e.g., p-xylene) by oxidation in an acetic acid solvent with oxygen-containing gas in the presence of a cobalt-manganese-bromine complex catalyst, to which nickel (e.g., nickel acetate) and carbon dioxide in appropriate amts. are added to increase catalyst activity. Nickel has a synergistic effect with carbon dioxide and maximizes the formation of the desired acid having the corresponding number of carboxylic groups to the number of alkyl groups in the reactant.

IT 100-21-0P, Terephthalic acid, preparation  
 RL: IMF (Industrial manufacture); SPN (Synthetic preparation); **PREP (Preparation)**  
 (method and catalyst system with Co and Mn and Br and Ni and CO<sub>2</sub> in acetic acid for preparing aromatic carboxylic acids from alkylaroms. by liquid-phase oxidation)

RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 3 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2002:825634 CAPLUS

DOCUMENT NUMBER: 138:287989

TITLE: A study on process parameters in liquid phase oxidation of p-xylene under the Co/Mn/Br-based catalyst systems

AUTHOR(S): Son, Young-Bae; Park, Kyung-Lyne; Yu, Tae-kong; Kim, Jung-Hi; Oh, In-Seok; Kim, Young-Ho; Yang, Hyun-Soo  
 CORPORATE SOURCE: Dep. Fine Chemicals Eng. Chem., Chungnam National Univ., S. Korea

SOURCE: Nonmunjip - Ch'unghnam Taehakkyo Sanop Kisul Yon'guso (2001), 16(2), 116-124  
 CODEN: NCTYEO

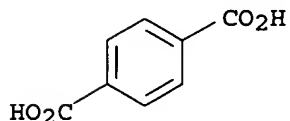
PUBLISHER: Ch'unghnam Taehakkyo Sanop Kisul Yon'guso

DOCUMENT TYPE: Journal

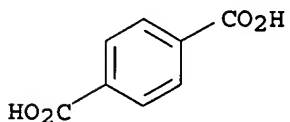
LANGUAGE: Korean

AB Liquid phase oxidation of p-xylene using mol. oxygen has been carried out on the Co/Mn/Br-based catalyst system in acetic acid as a solvent. Process parameters (total pressure, partial pressure of oxygen and temperature) and kinetics of the reaction were studied as a basic study in the viewpoint for the development of new catalyst system and process. The total pressure above 20 atm and the partial pressure of oxygen above 5 atm were required to keep the reactant in the liquid phase and to overcome the mass transfer limitation of oxygen. The oxidation rate and conversion were increased with increasing reaction temperature to the 100-190°C range. For a given initial concentration of p-xylene, the oxidation rate and the apparent activation energy were found to be of first-order and 4.24 kcal/mol, resp. The effect of the third components added to the Co/Mn/Br catalyst was also studied. It was found that Ni is only a good additive that can promote the catalyst performance.

IT 100-21-0P, Terephthalic acid, preparation  
 RL: SPN (Synthetic preparation); **PREP (Preparation)**  
 (product; study on process parameters in liquid phase oxidation of p-xylene  
 under Co/Mn/Br-based catalyst systems)  
 RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 4 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 2002:543886 CAPLUS  
 DOCUMENT NUMBER: 138:122875  
 TITLE: Combined Promotional Effect of CO<sub>2</sub> and Ni on  
 Co/Mn/Br Catalyst in the  
 Liquid-Phase Oxidation of p-Xylene  
 AUTHOR(S): Raju Burri, David; Jun, Ki-Won; Yoo, Jin S.; Lee, Chul  
 Wee; Park, Sang-Eon  
 CORPORATE SOURCE: Catalysis Center for Molecular Engineering, Korea  
 Research Institute of Chemical Technology, Yuseong,  
 Daejeon, 305-600, S. Korea  
 SOURCE: Catalysis Letters (2002), 81(3-4), 169-173  
 CODEN: CALEER; ISSN: 1011-372X  
 PUBLISHER: Kluwer Academic/Plenum Publishers  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB The promotional effects of CO<sub>2</sub> and Ni were studied on Co/  
 Mn/Br catalyst in the liquid-phase oxidation of p-xylene to  
 terephthalic acid using mol. oxygen as an oxidant and acetic acid as a  
 solvent individually as well as in combination. The enhanced activity of  
 Co/Mn/Br catalyst was observed on both CO<sub>2</sub> and Ni  
 promoters independently and also in combination. The activity enhancement  
 in the combination of CO<sub>2</sub> and Ni promoters on this catalyst is  
 found to be remarkable.  
 IT 100-21-0P, Terephthalic acid, preparation  
 RL: SPN (Synthetic preparation); **PREP (Preparation)**  
 (combined promotional effect of CO<sub>2</sub> and Ni on Co/Mn  
 /Br catalyst in liquid-phase oxidation of p-xylene)  
 RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

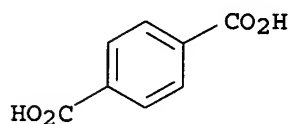
L7 ANSWER 5 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 2000:619623 CAPLUS  
 DOCUMENT NUMBER: 134:42462  
 TITLE: Formation and role of cobalt and manganese cluster  
 complexes in the oxidation of p-xylene  
 AUTHOR(S): Chavan, S. A.; Halligudi, S. B.; Srinivas, D.;

Ratnasamy, P.  
CORPORATE SOURCE: National Chemical Laboratory, Pune, 411008, India  
SOURCE: Journal of Molecular Catalysis A: Chemical ( 2000), 161(1-2), 49-64  
CODEN: JMCCF2; ISSN: 1381-1169  
PUBLISHER: Elsevier Science B.V.  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The mono- and multinuclear metal complexes present during the aerial oxidation of p-xylene by the homogeneous catalyst systems viz. Co/Br, Mn/Br, Co/Mn/Br, Co/Ce/Br, Co/Zr/Br, Co/Mn/Zr/Br, Co/Mn/Ce/Br, Ni/Mn/Br and Ni/Mn/Zr/Br in acetic acid solvent have been investigated by electronic and EPR spectroscopies. The reaction mixts. contain, in addition to Co(OAc)2·4H2O and Mn(OAc)2·4H2O, species like Co(OAc)Br, Co(OAc)3, Co3(O)(OAc)x, Mn3(O)(OAc)x and hetero-multinuclear complexes like Co2Mn(O)(OAc)x and CoMn2(O)(OAc)x. While mononuclear Co(OAc)2, Mn(OAc)2 and Co(OAc)Br complexes predominate in the initial stages of the oxidation reaction, significant concns. of multinuclear Co(III) and Mn(III) complexes are detected in the later stages. Zr(IV), when present, facilitates the oxidation of Mn(II) to Mn(III), a crucial step in the kinetic pathway of the oxidation reaction. EPR results indicate the presence of homo-nuclear Mn3(O)(OAc)x type clusters as the major species with a minor component of CoMn2(O)(OAc)x complex in catalyst systems with Co:Mn = 1:3 (mol). When an excess of cobalt catalyst is used (e.g., Co:Mn = 3:1) or when Zr and Br are also present in optimal concns., the heteronuclear cluster CoMn(O)(OAc) predominates while Co(O)(OAc) and CoMn(O)(OAc) occur as minor constituents. The yield of terephthalic acid (TA) is enhanced at high concns. of cluster complexes like Co2Mn(O)(OAc)x and CoMn2(O)(OAc)x. The combination Co/Mn/Zr (3:1:0.1 mol), exhibits a high catalytic activity and selectivity for terephthalic acid, especially in the presence of an optimal concentration of the bromide ion.

IT 100-21-0P, Terephthalic acid, preparation  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(formation and role of cobalt and manganese cluster complexes in oxidation of p-xylene to terephthalic acid)

RN 100-21-0 CAPLUS  
CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



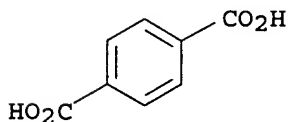
REFERENCE COUNT: 39 THERE ARE 39 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 6 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1997:130100 CAPLUS  
DOCUMENT NUMBER: 126:131886  
TITLE: Continuous process for the manufacture of terephthalic acid of monomer purity by liquid-phase catalytic oxidation of p-xylene  
INVENTOR(S): Vovk, Ludmila Stepanovna; Linnik, Vladimir Petrovich; Petrov, Viktor Pavlovich; Nazimok, Vladimir Filippovich; Tishchenko, Valery Ivanovich  
PATENT ASSIGNEE(S): Mogilev Order of Lenin Proizvodstvennoe Obiedinenie

SOURCE: "Khimvolokno", Belarus  
PCT Int. Appl., 23 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: Russian  
FAMILY ACC. NUM. COUNT: 2  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9641791	A1	19961227	WO 1996-BY4	19960508 <--

W: JP, KR, RU, US  
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE  
PRIORITY APPLN. INFO.: BY 1995-287 A 19950608  
OTHER SOURCE(S): MARPAT 126:131886  
AB The quality of terephthalic acid (I) is improved and unit consumption of AcOH (solvent) reduced by a combination of 2 interdependent processes for oxidation and recrystn. in a single aqueous AcOH solvent. The oxidation process is carried out in a cascade of 2 reactors arranged in series at temps. of 180-199°C in the presence of a catalyst comprising mixts. of Co, Mn, Ni, Na, Li and K salts and mixts. of HBr and alkali metal bromides or brominated alkanes, e.g., MeCHBrCH<sub>2</sub>Br, as sources of Br. Intermediate products are removed from I at the stage of recrystn. of I in AcOH under exptl. established conditions. The mother liquors are recirculated once the filtrates containing the raw (1st filtrate) and the pure (2nd filtrate) I have been separated during the 1st and 2nd oxidation stages, resp. A flow diagram of the process is included.  
IT 100-21-0P, Terephthalic acid, preparation  
RL: IMF (Industrial manufacture); PREP (Preparation)  
(continuous process for the manufacture of terephthalic acid of monomer purity by liquid-phase catalytic oxidation of p-xylene)  
RN 100-21-0 CAPLUS  
CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 7 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1996:598540 CAPLUS  
DOCUMENT NUMBER: 125:222740  
TITLE: Manufacture and purification of terephthalic acid with prevention of reactor corrosion  
INVENTOR(S): Hara, Toshitsuna; Kasai, Yoshinori; Ishimaru, Masaharu; Fukuda, Nobuo  
PATENT ASSIGNEE(S): Mitsubishi Chemical Corp., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 08193048	A2	19960730	JP 1995-5087	19950117 <--

PRIORITY APPLN. INFO.: JP 1995-5087 19950117  
AB The process comprises (1) oxidation of p-xylene in Br- and heavy

metal-containing AcOH, (2) purification by hydrogenation of aqueous terephthalic acid  
(I) with Pt-group catalysts, (3) crystallization of the purified I, and (4) drying

of wet I crystals in driers having Ni alloy parts in contact with the crystal heated at  $\geq 100^\circ$ . Oxidation of p-xylene in air in AcOH containing Co, Mn, and Br catalysts gave I, the aqueous I was fed into a distilling column containing Pd/C with H at  $290^\circ$ , crystallized by cooling to  $150^\circ$ , then dried in a dryer having Hastelloy C-lined parts (in contact with the crystal at  $110^\circ$ ). The dryer had no corrosion over 1 yr.

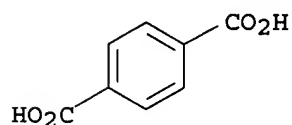
IT 100-21-0P, Terephthalic acid, preparation

RL: IMF (Industrial manufacture); PUR (Purification or recovery)  
; PREP (Preparation)

(manufacture and purification of terephthalic acid with reactor corrosion prevention by Ni alloy linings)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 8 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1995:606805 CAPLUS

DOCUMENT NUMBER: 123:9155

TITLE: Production method of high purity isomers of benzenedicarboxylic acids

INVENTOR(S): Nazimok, Vladimir Filippovich; Goncharova, Nadezhda Nikolaevna; Yurjev, Valerij Petrovich; Manzurov, Vladimir Dmitrievich

PATENT ASSIGNEE(S): Samsung General Chemicals Co., Ltd., S. Korea; Joint-Stock Company of Research and Design Institute of Monomers

SOURCE: PCT Int. Appl., 45 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9509143	A1	19950406	WO 1993-KR106	19931130 <--
W: AT, AU, BB, BG, BR, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KZ, LK, LU, MN, MW, NL, NO, NZ, PL, PT, RO, SD, SE, SK, UA, VN				
RW: BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
RU 2047594	C1	19951110	RU 1993-46190	19930928 <--
RU 2047595	C1	19951110	RU 1993-46191	19930928 <--
KR 9700136	B1	19970104	KR 1993-21276	19931014 <--
BR 9305996	A	19971021	BR 1993-5996	19931120 <--
CA 2128719	AA	19950329	CA 1993-2128719	19931130 <--
CA 2128719	C	19980609		
AU 9455763	A1	19950418	AU 1994-55763	19931130 <--
GB 2286588	A1	19950823	GB 1994-15915	19931130 <--
GB 2286588	B2	19960911		
ES 2081265	A1	19960216	ES 1994-50020	19931130 <--
ES 2081265	B1	19961016		
DE 4397599	T	19970724	DE 1993-4397599	19931130 <--



DE 4397599	C2	19980219		
RO 113850	B1	19981130	RO 1994-1218	19931130 <--
PL 175685	B1	19990129	PL 1993-308537	19931130 <--
JP 3009223	B2	20000214	JP 1994-516877	19931130 <--
JP 08506571	T2	19960716		
SK 280582	B6	20000410	SK 1994-877	19931130 <--
CN 1103860	A	19950621	CN 1994-100679	19940112 <--
CN 1050118	B	20000308		
BE 1008546	A4	19960604	BE 1994-702	19940726 <--
FR 2710638	A1	19950407	FR 1994-11253	19940921 <--
FR 2710638	B1	19960426		

PRIORITY APPLN. INFO.:

RU 1993-46190	A	19930928
RU 1993-46191	A	19930928
WO 1993-KR106	W	19931130

OTHER SOURCE(S): CASREACT 123:9155

AB An improved process for producing highly purified benzenedicarboxylic acid isomers, having color index of  $\leq 10^{\circ}\text{H}$  and containing  $< 0.0025\%$  carboxybenzyldehyde (CBA) impurity, without an addnl. catalytic reductive purification step, comprises (a) an oxidation step wherein xylene isomer is oxidized with mol. O or mol. O containing gas in the presence of a catalyst system composed of Co Mn, Br and at least one selected from Ni, Cr, Zr and Ce in low aliphatic carboxylic acid; and (b) an extraction/post-oxidation step wherein the oxidation product is crystalline to give a cake

of crude benzenedicarboxylic acid isomer, the cake is reslurried by adding lower aliphatic carboxylic acid solvent thereto followed by heating in order to extract impurities contained therein into the solvent, and the resulting slurry is oxidized with said catalyst system at a temperature of  $2-80^{\circ}$  lower than that of said heating, each of said oxidation and

extraction/post-oxidation

being carried out once or twice, provided that any one or both of said steps should be carried out twice. According to the invention, the solvent employed to extract impurities is recycled from the subsequent

oxidation

steps. P-xylene, AcOH, H<sub>2</sub>O, Co, Mn, Ni and Br were heated to  $160^{\circ}$ , oxidation effected at  $198^{\circ}$  and 19 kg/cm<sup>2</sup> for 40 min resulting in 20% terephthalic acid, 25 ppm 4-carboxybenzaldehyde and color index of  $8^{\circ}\text{H}$ .

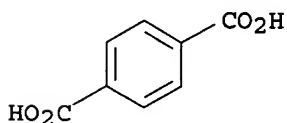
IT 100-21-0P, 1,4-Benzenedicarboxylic acid, preparation

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(production method of high purity isomers of benzenedicarboxylic acids)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 9 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1992:129688 CAPLUS

DOCUMENT NUMBER: 116:129688

TITLE: Developments in terephthalic acid manufacture

AUTHOR(S): Nazimok, V. F.; Pivovar, L. M.

CORPORATE SOURCE: USSR

SOURCE: Khimicheskije Volokna (1991), (6), 38-40

CODEN: KVLKA4; ISSN: 0023-1118

DOCUMENT TYPE: Journal

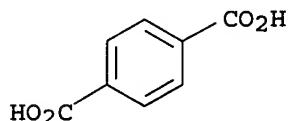
LANGUAGE: Russian

AB A scheme is presented for the continuous manufacture of terephthalic acid (I) by liquid-phase oxidation of p-xylene (II) in HOAc in the presence of a Co-Mn-Ni-Br catalyst. The oxidation is conducted in 3 steps. High-quality I can be obtained at 180-200° with 17% II in the starting mixture on using an optimized catalyst composition Aliphatic bromo C3-6-hydrocarbons are used as promoters.

IT 100-21-0P, Terephthalic acid, preparation  
 RL: IMF (Industrial manufacture); PREP (Preparation)  
 (manufacture of, by liquid-phase oxidn.of xylene, technol. for continuous)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 10 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1990:197849 CAPLUS

DOCUMENT NUMBER: 112:197849

TITLE: Continuous two-stage oxidation of aromatic hydrocarbons to aromatic carboxylic acids in an aqueous system

INVENTOR(S): Nowicki, Neal R.; Lowry, James D., Jr.

PATENT ASSIGNEE(S): Amoco Corp., USA

SOURCE: U.S., 5 pp.  
 CODEN: USXXAM

DOCUMENT TYPE: Patent  
 LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4892970	A	19900109	US 1985-814510	19851230 <--
PRIORITY APPLN. INFO.:			US 1985-814510	19851230

AB Benzenes (I) disubstituted with oxidizable substituents such as (hydroxy)alkyl, aldehyde, or carboalkyl are oxidized to their corresponding carboxylic acid derivs. in an aqueous solvent system in a continuous, 2-stage process comprising (1) partial (75-85%) oxidation of a feed stock containing I 100, H2O .apprx.5-100, ionic catalytic metal (Mn and at least one of Co, Ni, Zr or its mixture) 0.1-5.0 weight parts, and Br .apprx.10-300 atom % (based on the total catalytic metal) with a stoichiometric excess of an O-containing gas at .apprx.300-410°F and a pressure high enough to maintain a liquid phase, wherein byproduct formation is minimized and (2) addition of supplement Br to the first stage reaction mixture to raise the concentration of Br (50-500 atom % based on the metal) and of an O-containing gas at .apprx.400-480°F, wherein the added Br effectively suppresses decarboxylation reactions that typically occur at higher oxidation temperature Thus, p-xylene (II) was oxidized to terephthalic acid

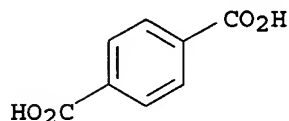
(III) in H2O in the presence of Co, Mn, and Br in the first stage at 380°F and 525 psig (8 psi O) using Co and Mn 2.7 weight%, Mn/Co (3.0 % of II), Br/Mn + Co (0.35), H2O/II (0.3) and in the second stage at 453°F and 510 psig (11 psi O) using Co and Mn (3.3% of II), Br/Mn + Co (2.8, 8 fold increase) and H2O/II (2.3). The slurry obtained contained toluic acid 0.08, 4-(HO2C)C6H4CHO 0.18, III 33.0, and high mol.

weight component 0.20 weight %.

IT 100-21-0P, 1,4-Benzenedicarboxylic acid, preparation  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of, by oxidation of p-xylene)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 11 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1989:192438 CAPLUS

DOCUMENT NUMBER: 110:192438

TITLE: Oxidation process for the manufacture of aromatic acids from alkylaromatic compounds

INVENTOR(S): Partenheimer, Walter; Schammel, Wayne P.

PATENT ASSIGNEE(S): Amoco Corp., USA

SOURCE: U.S., 5 pp.  
 CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

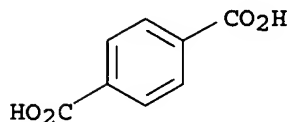
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4786753	A	19881122	US 1987-50860	19870518 <--
EP 362443	A1	19900411	EP 1988-309255	19881005 <--
EP 362443	B1	19940112		
R: BE, DE, ES, FR, GB, IT, NL				
ES 2047557	T3	19940301	ES 1988-309255	19881005 <--
JP 02138149	A2	19900528	JP 1988-263044	19881020 <--
JP 2746346	B2	19980506		
CN 1042534	A	19900530	CN 1988-107608	19881105 <--
CN 1024660	B	19940525		
PRIORITY APPLN. INFO.:			US 1987-50860	19870518
			EP 1988-309255	A 19881005

OTHER SOURCE(S): CASREACT 110:192438

AB A process of oxidizing di- and trimethylbenzenes with O<sub>2</sub> to benzenedi- and -tricarboxylic acids in the liquid phase in the presence of a C<5 aliphatic acid, H<sub>2</sub>O, or a mixture of the aliphatic acid and H<sub>2</sub>O at .apprx.100-260° and at a pressure to maintain as liquid phase 70-80% of the reaction medium comprised conducting said oxidation in the presence of a catalyst system comprising a source of Br with Ni, Zr, and Mn wherein for each g-mol of p-xylene, m-xylene, or pseudocumene in the oxidation there is from .apprx.4 to .apprx.20 mg-atom Ni, .apprx.0.10 to .apprx.0.30 mg-atom Zr, .apprx.2 to .apprx.10 mg-atom total Mn, and from .apprx.8 to 24 mg-atom Br. The addition of Ni and Zr to oxidation catalysis provided by heavy, transition metal-Br ion combination containing at least Mn ion uniquely increases catalytic activity of said combination for converting Me groups to CO<sub>2</sub>H on the benzene nucleus and enables the elimination of the expensive catalyst Co. The preparation of phthalic acid, isophthalic acid, and trimellitic acid from p-Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m-Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, and pseudocumene, resp., using various Ni, Zr, Mn, and Br combinations is given.

IT 100-21-0P, Terephthalic acid, preparation  
 RL: SPN (Synthetic preparation); PREP (Preparation)

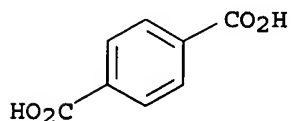
(preparation of, by catalytic oxidation of p-xylene)  
RN 100-21-0 CAPLUS  
CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 12 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1986:68602 CAPLUS  
DOCUMENT NUMBER: 104:68602  
TITLE: Tere- or isophthalic acids  
INVENTOR(S): Nazimok, V. F.; Golubev, G. S.; Boyarkin, M. A.;  
Manzurov, V. D.; Yur'ev, V. P.; Khomin, V. V.;  
Pakhorukov, V. A.  
PATENT ASSIGNEE(S): All-Union Scientific-Research and Design Institute of  
Monomers, USSR  
SOURCE: U.S.S.R. From: Otkrytiya, Izobret. 1985, (29), 102.  
CODEN: URXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Russian  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SU 1171452	A1	19850807	SU 1983-3620406	19830510 <--

PRIORITY APPLN. INFO.: SU 1983-3620406 19830510  
AB Tere- and isophthalic acids are prepared by liquid-phase oxidation of p- or m-xylene, resp., with a gas containing mol. O in HOAc; in the 1st stage at 200-215°/20-26 atm gage in the presence of Co, Mn, or Ni salts and Br compds., with subsequent treatment of the reaction material, in the 2nd stage, at 180-200°/8.2-1.0 atm gage with steam and gas mixture The reaction mixture was further treated with 0.3-0.6 parts by weight of a reflux solution from crystallization zones, containing HOAc  
80-88, H2O 10-16, and ROR1 (R, R1 = H, Me, MeO) 1.5-4.0%.  
IT 100-21-0P, preparation  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of, by oxidation of xylene)  
RN 100-21-0 CAPLUS  
CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 13 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1980:567949 CAPLUS  
DOCUMENT NUMBER: 93:167949  
TITLE: Method of preparing terephthalic acid  
INVENTOR(S): Nazimok, V. F.; Kulakov, V. N.; Manzurov, V. D.;  
Boyarkin, M. A.; Golubev, G. S.; Simonova, T. A.;  
Valieva, R. A.; Petrov, A. A.; Zernov, P. N.; et al.

PATENT ASSIGNEE(S): All-Union Scientific-Research and Design Institute of Monomers, USSR  
 SOURCE: U.S.S.R. From: Otkrytiya, Izobret., Prom. Obraztsy, Tovarnye Znaki 1980, (21), 135.  
 CODEN: URXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Russian  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SU 739062	T	19800605	SU 1978-2591177	19780316 <--

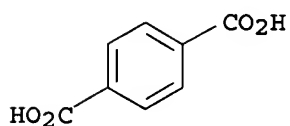
PRIORITY APPLN. INFO.: SU 1978-2591177 A 19780316

AB The title compound was prepared by the liquid phase oxidation of p-xylene in AcOH in presence of a Co, Mn, Ni catalyst and Br-initiator. The process was carried out in two stages. The first stage was at 210-30° and 24-7 atms in presence of a catalyst with total concentration of Co and Mn 0.045-0.15% of the reaction mixture and weight concentration of Ni 0.0005-0.005% and ratio of Mn:Ni = 30-100. The second stage was at 160-210° and 5-10 atms. The mixture contained 40-70% AcOH, 10-20% H2O, 15-50% N and 0.1-1.5% O.

IT 100-21-0P, preparation  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of, by oxidation of xylene)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 14 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1980:446199 CAPLUS

DOCUMENT NUMBER: 93:46199

TITLE: Recovery of nondissociated bromine in spent oxidation catalyst solution after terephthalic acid production

INVENTOR(S): Takeuchi, Hiroshi; Saeki, Satoshi

PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
 CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

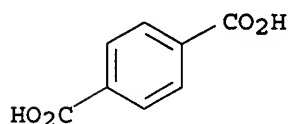
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 55027037	A2	19800226	JP 1978-99101	19780816 <--

PRIORITY APPLN. INFO.: JP 1978-99101 A 19780816

AB p-Xylene is oxidized with O in AcOH containing heavy-metal bromide, and the mother liquor is distilled first to recover AcOH and then at P mm Hg pressure and t° temperature to satisfy  $\log P \leq 9.57 - 3330/(t + 348)$ . Thus, AcOH 20 kg containing CoBr<sub>2</sub>·6H<sub>2</sub>O 20 and Mn(OAc)·2.4H<sub>2</sub>O 20 g in a Ti autoclave was injected at 200° and 20 kg/cm<sup>2</sup> gage with 20 kg/L 10% p-xylene and 5 kg/h air for 5 h, the product was collected continuously in a crystallization vessel at 180° and 10 kg/cm<sup>2</sup> gage and separated. The 90 L mother liquor containing some p-(HO<sub>2</sub>C)<sub>2</sub>C<sub>6</sub>H<sub>4</sub> (I), H<sub>2</sub>O 5%, Co

750, **Mn** 700, Fe 52, Cr 13, **Ni** 15, **Br**- 620, and undissocd. **Br** 1800 ppm was fed at 8 kg/h at the top of a distillation column filled with Raschig rings at 114° to sep. AcOH and H<sub>2</sub>O. The 1 kg solution from the bottom was fed 1 kg/h into an Arther Smith thin-film evaporator at 100°/1 mm Hg to recover 92% **Br**, vs. 7.3% at 110°/250 mm Hg. When recycled along with Co and **Mn** carbonates, the I yield containing 190-210 ppm 4-HCOC<sub>6</sub>H<sub>6</sub>CO<sub>2</sub>H was 96-8% in 6 cycles.

IT 100-21-0P, preparation  
 RL: SPN (Synthetic preparation); **PREP** (Preparation)  
 (preparation of, by oxidation of p-xylene)  
 RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



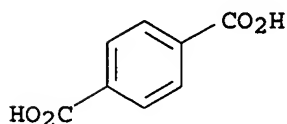
L7 ANSWER 15 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1979:204824 CAPLUS  
 DOCUMENT NUMBER: 90:204824  
 TITLE: Recovery of liquid-phase oxidation catalyst and solvent  
 INVENTOR(S): Miyake, Tetsuya; Takeuchi, Hiroshi; Tauchi, Masatoshi; Saeki, Miciyuki; Saka, Kazuki  
 PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 53104590	A2	19780911	JP 1977-19667	19770224 <--

PRIORITY APPLN. INFO.: JP 1977-19667 A 19770224

AB In the oxidation of p-xylene (I) [106-42-3] with mol. O to terephthalic acid (I) [100-21-0] in the presence of catalysts containing Co, **Mn**, and **Br** in lower aliphatic monocarboxylic acid, the reaction mixture after separation of II was treated with **Br**--type anion exchanger to remove Co and **Mn** ions and Cu and Zn ions if present, and the metal compds. were recovered and recycled. The effluent from the anion exchanger treatment was treated with a carboxylate-type, weakly basic anion exchanger at 20-150° to remove **Br** ion and **Ni** if present, which were also recovered and recycled. For example, a mixture of 20 kg AcOH [64-19-7], 20 g (as Co) CoBr<sub>2</sub>·6H<sub>2</sub>O and 20 g (as **Mn**) **Mn**(OAc)·2.4H<sub>2</sub>O at 200°/20 kg/cm<sup>2</sup> gage was fed with 10% I solution in AcOH at the rate of 20 g/h and 20 kg/h air, maintaining the reaction mixture at 180°/10 kg/cm<sup>2</sup> gage for 5 h, and the reaction mixture was continuously discharged, separated from II, and passed through a pyridinium bromide-type anion exchanger to give an effluent (III) containing Co <1, **Mn** <1, and **Br** 2400 ppm, compared with 920, 910, and 2700, resp., for the reaction mixture. The spent resin was eluted with AcOH containing 2% water to give a 30 mL solution containing Co 15,000, **Mn** 14,500, and **Br** 4200 ppm. III passed through a pyridinium acetate-type anion-exchange column at 80° to give an effluent containing <10 ppm **Br**; elution of the resin with AcOH gave a solution containing 40,000 ppm **Br**. AcOH was recovered in

93.8% yield by fractional distillation  
 IT 100-21-0P, preparation  
 RL: IMF (Industrial manufacture); **PREP (Preparation)**  
 (manufacture of, from xylene, recovery and recycle of solvent and oxidation catalysts in)  
 RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)

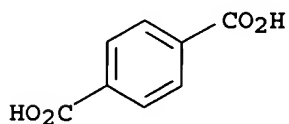


L7 ANSWER 16 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1978:604884 CAPLUS  
 DOCUMENT NUMBER: 89:204884  
 TITLE: Recovery of cobalt-manganese-bromide catalyst  
 INVENTOR(S): Miyake, Tetsuya; Takeuchi, Hiroshi; Tauchi, Masatoshi;  
 Saeki, Michiyuki; Saka, Kazuki  
 PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 53102290	A2	19780906	JP 1977-16756	19770218 <--
PRIORITY APPLN. INFO.:			JP 1977-16756	A 19770218

AB With Co and Mn catalyst and Br as accelerator, alkylbenzene or its derivative is oxidized with O in lower fatty acid solvent into the corresponding carboxylic acid, the resulting solution is contacted with anion-exchange resin containing pyridine ring in the bromide or solvent anion form, and eluted to recover Co and Mn with or without Br. Thus, p-xylene was oxidized in AcOH containing Co, Mn, and Br in a 2 L Ti vessel at 205° and 15 atm, cooled to 100°, and filtered to sep. terephthalic acid; the 500 mL solution containing p-xylene 50, 4-carboxybenzaldehyde 450, p-cresol 20 ppm, Co 0.10, Mn 0.02, Br 0.20, Fe 0.005, Cr 0.003, and NO 0.002% was passed through a pyridine anion exchanger column 12 diameter + 100 mm at 80° and 200 mL/h. The effluent contained Co, Mn <0.001 each, Br <0.002, Fe 0.0049, Cr 0.0028, and Ni 0.0017%, vs. 0.072, 0.012, 0.033, 0.0047, 0.0029, and 0.0019% with Dowex 1X4.

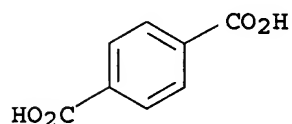
IT 100-21-0P, preparation  
 RL: **PREP (Preparation)**  
 (manufacture of, by oxidation of xylene, recovery of cobalt-manganese catalysts for)  
 RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 17 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1977:106180 CAPLUS  
 DOCUMENT NUMBER: 86:106180  
 TITLE: High-purity terephthalic acid  
 INVENTOR(S): Namie, Koushi; Takeda, Shinichi  
 PATENT ASSIGNEE(S): Teijin Hercules Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 51108028	A2	19760925	JP 1975-31829	19750318 <--
JP 59008253	B4	19840223		

PRIORITY APPLN. INFO.: JP 1975-31829 A 19750318  
 AB Liquid-phase catalytic oxidation of p-xylene to terephthalic acid with O in a carboxylic acid solvent was carried out at 170-280° with 500-6000 ppm Mn + Ni concentration in the solvent and ≥0.8 time weight of Br (as HBr) based on the total metal amount Thus, 200 g AcOH containing 0.446 g Mn(OAc)2, 0.424 g Ni(OAc)2, and 1.3 g 47% HBr [0.1% 1:1 Mn-Ni in AcOH, Br/(Mn + Ni) = 3] was treated with 25 g/h p-xylene and 1.5 L./min air at 220°/20 kg/cm2 in an autoclave to give 97% terephthalic acid containing 190 ppm p-carboxybenzaldehyde, vs. 92% and 1240 ppm, resp., with 0.22 g 47% HBr.  
 IT 100-21-0P, reactions  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of, from p-xylene)  
 RN 100-21-0 CAPLUS  
 CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)



L7 ANSWER 18 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1975:64116 CAPLUS  
 DOCUMENT NUMBER: 82:64116  
 TITLE: Recovery of heavy metal bromides and hydrogen bromide from reaction mixture for producing terephthalic acid  
 INVENTOR(S): Shigeyasu, Mottoo; Ozaki, Takeo; Kusano, Nobuo  
 PATENT ASSIGNEE(S): Matsuyama Petrochemicals Inc.  
 SOURCE: Ger. Offen., 38 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 2  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2415393	A1	19741024	DE 1974-2415393	19740329 <--
DE 2415393	C3	19791018		
DE 2415393	B2	19790301		
JP 49123192	A2	19741125	JP 1973-36351	19730330 <--



JP 55007300	B4	19800223		
JP 54019399	B4	19790714	JP 1973-47826	19730426 <--
JP 49133290	A2	19741220		
BE 813080	A1	19740715	BE 1974-142667	19740329 <--
PRIORITY APPLN. INFO.:			JP 1973-36351	A 19730330
			JP 1973-47826	A 19730426

AB **Br**, **Co**, and **Mn** are recovered from the residue obtained after the removal of terephthalic acid (**I**) and the remaining solvent from the reaction mix for the preparation of **I** by oxidation of p-dialkylbenzene in the presence of brominated heavy metal catalysts, particularly **Co** and **Mn**, by treating the residue with H<sub>2</sub>O and O<sub>2</sub> in the presence of a sulfide, such as H<sub>2</sub>S, Na<sub>2</sub>S, NaHS, K<sub>2</sub>S, NH<sub>4</sub>HS, etc., separating precipitated insol.

impurities which may include Fe and Cr oxides and sulfides of Pb, Mo, Cu, Ni, or Cd, treating the aqueous solution with a strong acid cation exchange resin in H<sup>+</sup> form, to absorb the **Co** and **Mn** ions, and distilling the remaining solution to recover HBr. The absorbed **Co** and **Mn** ions are removed from the ion exchange resin by eluting with aqueous HBr and the eluant distilled to recover excess HBr and isolate bromides of **Co** and **Mn**. Thus, to a tarry residue 700 recovered after the preparation of **I** with AcOH solvent, **CoBr**<sub>2</sub>, **Mn** acetate, and HBr and after removal of **I** and AcOH, which residue contains **Co** 4.6, **Mn** 0.27, Fe 0.32, Cr 0.09, **Br** 4.5%, Cu 12, Mo 6, and Pb 4 ppm, H<sub>2</sub>O 1050 kg is added, heated to 70° for 2 hr with stirring and 300 l./hr of air together with 40 l. of H<sub>2</sub>S passed through, passed to a crystallization vessel where it is cooled to 30°, centrifuged to remove solids to provide 950 kg of filtrate containing **Co** 3, **Mn** 0.15, **Br** 3%, as well as Fe 59, Cr 5.4, Cu 1.5, Mo 0.7, and Pb 0.5 ppm. The yield of **Co** extracted is 89%. The **Co** and **Mn** ions are absorbed in an ion exchange resin column. When the resin has absorbed its capacity of metal ions, it is 1st washed with H<sub>2</sub>O (the resultant H<sub>2</sub>O being used for the next extraction step), then washed with 10% AcOH to remove organic impurities, and

then eluted with 3N HBr to desorb **Co** and **Mn**; the resultant eluant containing **CoBr**<sub>2</sub>, **MnBr**<sub>2</sub>, and HBr, is distilled recovering excess HBr and leaving a product containing **Co** 23.8, **Mn** 1.2, Fe 0.04, Cr 0.004, **Br** 74.6%, Cu 3, Mo 3, and Pb 3 ppm. The yield of **Co** from the extract is 97%.

IT 100-21-0P, preparation

RL: IMF (Industrial manufacture); PREP (Preparation)  
(waste water from manufacture of, hydrogen bromide and metal bromide recovery from, ion exchange and distillation in)

RN 100-21-0 CAPLUS

CN 1,4-Benzenedicarboxylic acid (9CI) (CA INDEX NAME)

